(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 982 628 A2

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
01.03.2000 Bulletin 2000/09

(51) Int. Cl.[7]: G03F 7/004

(21) Application number: 99116705.7

(22) Date of filing: 25.08.1999

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: 26.08.1998 JP 24014398

(71) Applicant:
Sumitomo Chemical Company, Limited
Chuo-ku Osaka 541-8550 (JP)

(72) Inventors:
- Fujishima, Hiroaki
  Toyonaka-shi, Osaka (JP)
- Uetani, Yasunori
  Toyonaka-shi, Osaka (JP)
- Araki, Kaoru
  Kyoto (JP)

(74) Representative:
VOSSIUS & PARTNER
Siebertstrasse 4
81675 München (DE)

(54) A chemical amplifying type positive resist composition

(57) A chemical amplifying type positive resist composition, excellent in adhesion to a substrate and good in resist performances and suitable for exposure using a KrF excimer laser, ArF excimer laser, or the like, which comprises a resin having a polymerization unit of 2-alkyl-2-adamantyl (meth)acrylate and a polymerization unit of a monomer selected from 3-hydroxy-1-adamantyl (meth)acrylate and (meth)acrylonitrile, and an acid generator is provided.

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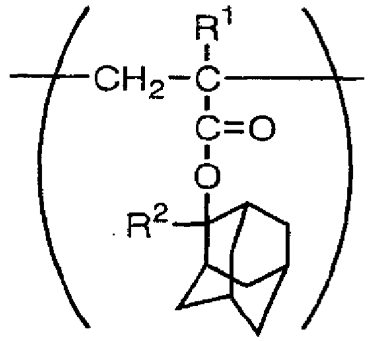
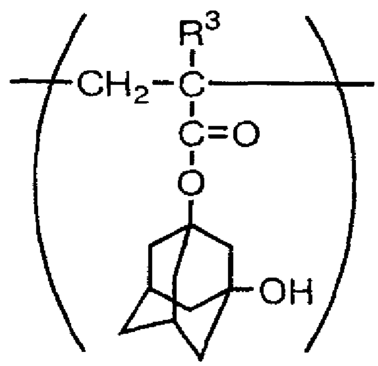


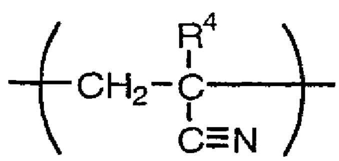
(I)

wherein R^1 represents hydrogen or methyl, and R^2 represents alkyl.



(II)

wherein R^3 represents hydrogen or methyl.



(III)

wherein R^4 represents hydrogen or methyl.

[0011] Therefore, the resin(1) has the unit of the above-described formula (I), and one or both of the units of the above-described formulae (II) and (III). The resin (1) may also have a polymerization unit of α-methacryloyloxy-γ-butyrolactone or a polymerization unit of maleic anhydride. The polymerization unit of α-methacryloyloxy-γ-butyrolactone herein cited means a unit in the polymer formed by the polymerization of α-methacryloyloxy-γ-butyrolactone. The polymerization unit of maleic anhydride means a unit in the polymer formed by the polymerization of maleic anhydride. These units are represented by the following formulae (IV) and (V), respectively:

formulae (II), (III), (IV), and (V).

[0014] The resin(1) has the polymerization unit of 2-alkyl-2-adamantyl (meth)acrylate represented by the formula (I), and one or both of the polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate represented by the formula (II) and the polymerization unit of (meth)acrylonitrile represented by the formula (III). The resin(1) may optionally have the polymerization unit of α-methacryloyloxy-γ-butyrolactone represented by the formula (IV) and/or the polymerization unit of maleic anhydride represented by the formula (V). Therefore, the resin(1) can be produced by conducting a copolymerization of 2-alkyl-2-adamantyl (meth)acrylate, and one or both of 3-hydroxy-1-adamantyl (meth)acrylate and (meth)acrylonitrile, and optionally α-methacryloyloxy-γ-butyrolactone and/or maleic anhydride.

[0015] Among these monomers, 2-alkyl-2-adamantyl (meth)acrylate can generally be produced by the reaction of 2-alkyl-2-adamantanol or a metallic salt thereof and acrylic acid halide or methacrylic acid halide. 3-Hydroxy-1-adamantyl (meth)acrylate can be produced by hydrolyzing 1,3-dibromoadamantane to prepare 1,3-dihydroxyadamantane which is then reacted with acrylic acid, methacrylic acid or halide thereof.

[0016] The polymerization unit of 2-alkyl-2-adamantyl (meth)acrylate represented by the formula (I) ensures the transmittance of a resist and contributes to the improvement of dry etching resistance due to the presence of an adamantane ring. Further, the 2-alkyl-2-adamantyl in this unit is cleaved by the action of an acid, and hence this unit contributes to the enhancement of alkali-solubility after exposure of a resist film. R^2 in the formula (I) is alkyl. This alkyl may have, for example, about 1 to 8 carbon atoms. In general, the alkyl is advantageously straight chain, but it may be branched when the number of carbons is 3 or more. Examples of R^2 include methyl, ethyl, n-propyl, isopropyl, and n-butyl. Among these examples, methyl or ethyl, particularly ethyl, is preferred as R^2 for the improvement of adhesion between a resist and a substrate, and for the improvement of resolution. A resist comprising the resin(1) of formula (I) wherein R^1 is methyl and R^2 is ethyl exhibits particularly high adhesion to a substrate.

[0017] The resin(2) has the polymerization unit of 2-ethyl-2-adamantyl methacrylate represented by the formula (Ia), and at least one of the polymerization units selected from the polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate represented by the formula (II), the polymerization unit of (meth)acrylonitrile represented by the formula (III), the polymerization unit of α-methacryloyloxy-γ-butyrolactone represented by the formula (IV), and the polymerization unit of maleic anhydride represented by the formula (V). Therefore, the resin(2) can be produced by conducting a copolymerization of 2-ethyl-2-adamantyl methacrylate and at least one of the monomers selected from 3-hydroxy-1-adamantyl (meth)acrylate, (meth)acrylonitrile, α-methacryloyloxy-γ -butyrolactone, and maleic anhydride.

[0018] 2-Ethyl-2-adamantyl methacrylate can be produced by the reaction of 2-ethyl-2-adamantanol or a metallic salt thereof, and methacrylic acid halide. For example, ethyl lithium is allowed to react with 2-adamantanone to form lithium 2-ethyl-2-adamanetanolate. The resulting lithium 2-ethyl-2-adamanetanolate is subjected to condensation with methacrylic acid halide to obtain 2-ethyl-2-adamantyl methacrylate.

[0019] The polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate represented by the formula (II), the polymerization unit of (meth)acrylonitrile represented by the formula (III), the polymerization unit of α-methacryloyloxy-γ-butyrolactone represented by the formula (IV), and the polymerization unit of maleic anhydride represented by the formula (V) have high polarity. The presence of any of these polymerization units in a resin(2) contributes to the improvement of the adhesion of a resist comprising the resin(2) to a substrate. Particularly, the polymerization unit of α-methacryloyloxy-γ-butyrolactone of formula (IV) is excellent in effect of adhesion improvement. The polymerization unit of α-methacryloyloxy-γ-butyrolactone exhibits a remarkable effect in combination with the polymerization unit of 2-ethyl-2-adamantyl methacrylate represented by the formula (Ia).

[0020] The polymerization unit of 2-alkyl-2-adamantyl (meth)acrylate of formula (I) also exhibits a remarkable effect in combination with the polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate or the polymerization unit of (meth)acrylonitrile. Further, among the above-described units, the polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate, the polymerization unit of (meth)acrylonitrile and the polymerization unit of maleic anhydride also contribute to the improvement of the dry etching resistance of a resist. Whereas the polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate and the polymerization unit of α-methacryloyloxy-γ-butyrolactone also contribute to the improvement of the resolution of the resist.

[0021] A resin for a chemical amplifying type positive resist itself is generally alkali-insoluble or alkali-slightly soluble However, a part of a group therein is cleaved by the action of an acid, and the resin becomes alkali-soluble after the cleavage. In the resin(1) or (2) specified in the present invention, 2-alkyl-2-adamantyl in the formula (I) or 2-ethyl-2-adamantyl in the formula (Ia) is cleaved by the action of an acid. Therefore, the presence of the polymerization unit of the formula (I) or (Ia) in the resin(1) or (2) causes a resist composition comprising the resin to act as a positive type. If required, the resin may also comprises other polymerization units having a group which are cleaved by the action of an acid.

[0022] Examples of the group in the other polymerization units which are cleaved by the action of an acid include various esters of carboxylic acid. Examples of the esters of carboxylic acid include alkyl esters such as tert-butyl ester, acetal type esters such as methoxymethyl ester, ethoxymethyl ester, 1-ethoxyethyl ester, 1-isobutoxyethyl ester, 1-iso-

2,4,6-tris(trichloromethyl)-1,3,5-triazine,
2-phenyl-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-chlorophenyl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-methoxyphenyl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-methoxy-1-naphthyl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(benzo[d][1,3]dioxolane-5-yl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-methoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(3,4,5-trimethoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(3,4-dimethoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(2,4-dimethoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(2-methoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-butoxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
2-(4-pentyloxystyryl)-4,6-bis(trichloromethyl)-1,3,5-triazine,
1-benzoyl-1-phenylmethyl p-toluenesulfonate (so-called benzointosylate),
2-benzoyl-2-hydroxy-2-phenylethyl p-toluenesulfonate (so-called α-methylolbenzointosylate),
1,2,3-benzenetriyl trismethanesulfonate,
2,6-dinitrobenzyl p-toluenesulfonate,
2-nitrobenzyl p-toluenesulfonate,
4-nitrobenzyl p-toluenesulfonate,
diphenyl disulfone,
di-p-tolyl disulfone,
bis(phenylsulfonyl)diazomethane,
bis(4-chlorophenylsulfonyl)diazomethane,
bis(p-tolylsulfonyl)diazomethane,
bis(4-tert-butylphenylsulfonyl)diazomethane,
bis(2,4-xylylsulfonyl)diazomethane,
bis(cyclohexylsulfonyl)diazomethane,
(benzoyl) (phenylsulfonyl)diazomethane,
N-(phenylsulfonyloxy)succinimide,
N-(trifluoromethylsulfonyloxy)succinimide,
N-(trifluoromethylsulfonyloxy)phthalimide,
N-(trifluoromethylsulfonyloxy)-5-norbornene-2,3-dicarboxyimide,
N-(trifluoromethylsulfonyloxy)naphthalimide and
N-(10-camphorsulfonyloxy)naphthalimide.

[0027] Generally in a chemical amplifying type positive resist composition, performance deterioration due to the deactivation of an acid associated with leaving after exposure can be reduced by adding basic compounds, especially basic nitrogen-containing organic compounds such as amines as quenchers. It is also preferable in the present invention that such basic compounds are added. Examples of the basic compounds to be used as quenchers include the ones represented by the following formulae:

be used alone or in combination of two or more thereof.

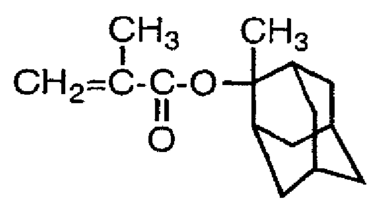
[0030] The resist film applied on a substrate and dried is subjected to an exposure treatment for patterning. Then, after a heat-treatment for promoting a protecting deblocking reaction, development by an alkali developer is conducted. The alkali developer herein used can be various kinds of alkaline aqueous solution used in this field. An aqeous solution of tetramethylammoniumhydroxide or (2-hydroxyethyl)trimethylammoniumhydroxide (so-called colline hydroxide) is generally used.

Examples

[0031] The present invention will be described in more detail by way of examples, which should not be construed as limiting the scope of the present invention. All parts in examples are by weight unless otherwise stated. The weight-average molecular weight is a value determined from gel permeation chromatography using polystyrene as a reference standard.

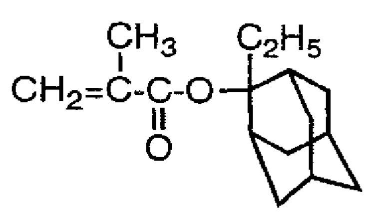
Monomer Synthesis Example 1 (synthesis of 2-methyl-2-adamantyl methacrylate)

[0032] 83.1 g of 2-methyl-2-adamantanol and 101 g of triethylamine were charged, and 200 g of methyl isobutyl ketone was added thereto to prepare a solution. Then, 78.4 g of methacrylic acid chloride (1.5 mole times with respect to 2-methyl-2-adamantanol) was added dropwise thereto, followed by stirring at room temperature for 10 hours. After filtration, the organic layer was washed with a 5 % by weight aqueous solution of sodium bicarbonate, followed by washing with water for 2 times. The organic layer was concentrated, and then subjected to distillation under reduced pressure to obtain 2-methyl-2-adamantyl methacrylate represented by the following formula in a 75 % yield.

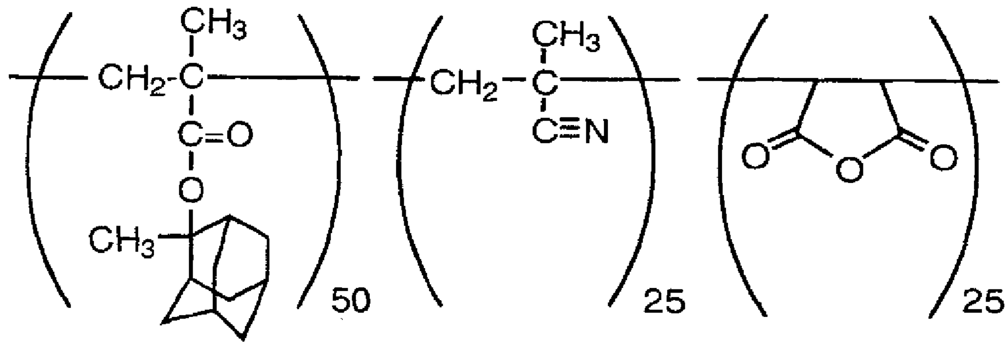


Monomer Synthesis Example 2 (synthesis of 2-ethyl-2-adamantyl methacrylate)

[0033] 50 g of diethyl ether was added to 31.1 g of 2-adamantanone to prepare a solution. Then, 200 ml of a diethyl ether solution containing ethyl lithium in a concentration of 1.14 mole/L was added dropwise thereto at such a rate as to keep the temperature of the solution not exceeding 10 °C. After stirring the solution thus obtained at 0 °C for 2 hours, 26.2 g of methacrylic acid chloride (1.2 mole times with respect to 2-adamantanone) was added dropwise thereto at such a rate as to keep the temperature not exceeding 10 °C. After the completion of dropwise addition, the resulting solution was stirred at room temperature for 12 hours. Thereafter, the deposited inorganic salts were separated by filtration, and the organic layer was washed with a 5 % by weight aqueous solution of sodium bicarbonate, followed by washing with water for 2 times. The organic layer was concentrated, and then subjected to distillation under reduced pressure to obtain 2-ethyl-2-adamantyl methacrylate represented by the following formula in a 60 % yield.



the operation of pouring the reaction solution into a large amount of heptane to cause precipitation was repeated 3 times, thus the solution was purified. As a result, a copolymer represented by the following formula, and having a composition mole ratio of each unit of 50 : 25 : 25, and having a weight-average molecular weight of about 8,000 was obtained. The resulting copolymer is referred to as resin B.

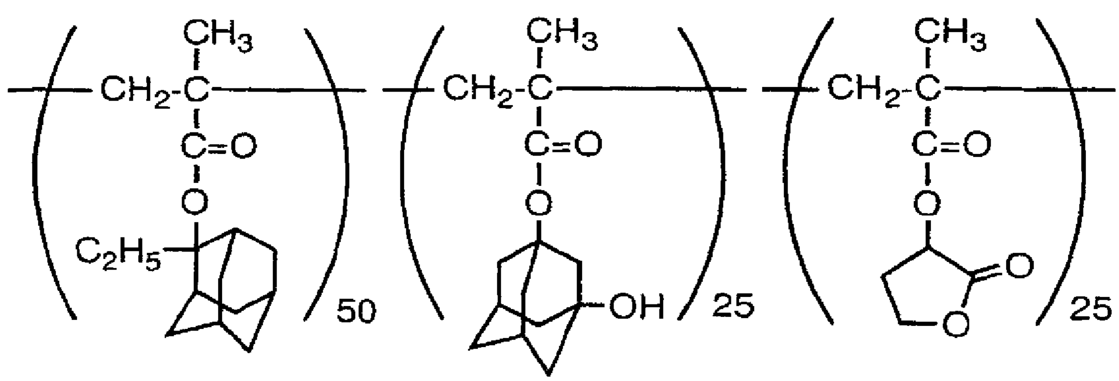


Resin Synthesis Example 2-2

[0037] Similarly, 2-methyl-2-adamantyl methacrylate, methacrylonitrile and α-methacryloyloxy-γ-butyrolactone are copolymerized to give a ternary copolymer having their respective polymerization units.

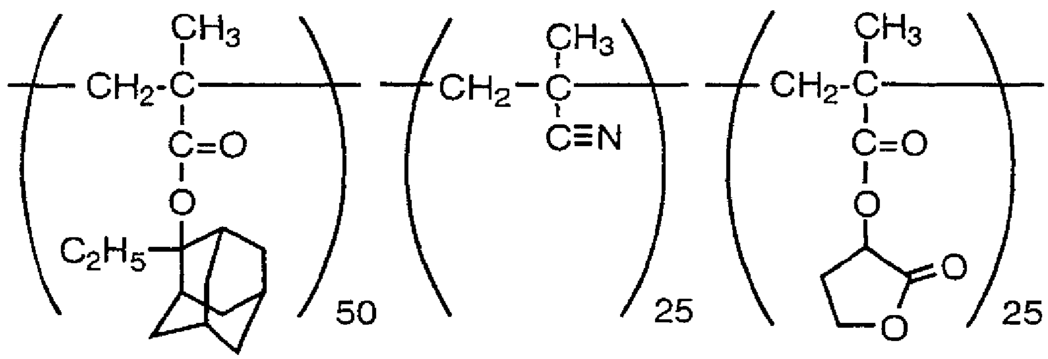
Resin Synthesis Example 3 (synthesis of resin C)

[0038] The same operations as in the resin synthesis example 1 were conducted, except that 2-methyl-2-adamantyl methacrylate, 3-hydroxy-1-adamantyl methacrylate and α-methacryloyloxy-γ-butyrolactone were replaced with 2-ethyl-2-adamantyl methacrylate, 3-hydroxy-1-adamantyl methacrylate and α-methacryloyloxy-γ-butyrolactone in a mole ratio of 5 : 2.5 : 2.5 (20.0g : 9.5g : 7.3g), respectively. As a result, a copolymer represented by the following formula, and having a composition mole ratio of each unit of 50 : 25 : 25, and having a weight-average molecular weight of about 9,200 was obtained. The resulting copolymer is referred to as resin C.



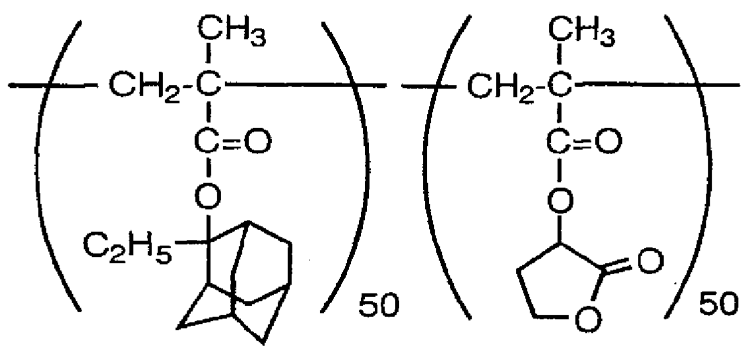
Resin Synthesis Example 4 (synthesis of resin D)

[0039] The same operations as in the resin synthesis example 2 were conducted, except that 2-methyl-2-adamantyl



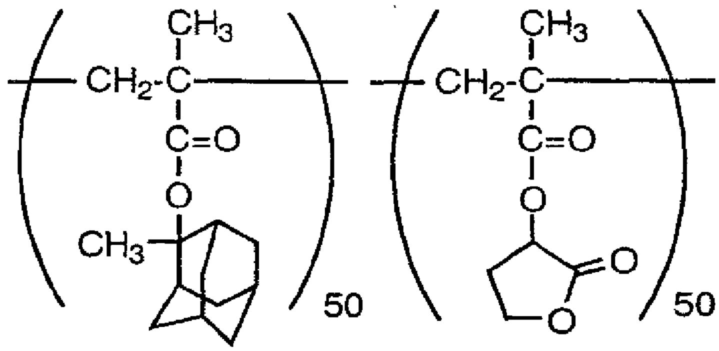
Resin Synthesis Example 7 (synthesis of resin G)

[0042] The same operations as in the resin synthesis example 1 were conducted, except that 2-methyl-2-adamantyl methacrylate, 3-hydroxy-1-adamantyl methacrylate and α-methacryloyloxy-γ-butyrolactone were replaced with 2-ethyl-2-adamantyl methacrylate and α-methacryloyloxy-γ-butyrolactone in a mole ratio of 5 : 5 (40.0 g : 29.3 g) , respectively. As a result, a copolymer represented by the following formula, and having a composition mole ratio of each unit of 50 : 50, and having a weight-average molecular weight of about 5,600 was obtained. The resulting copolymer is referred to as resin G.



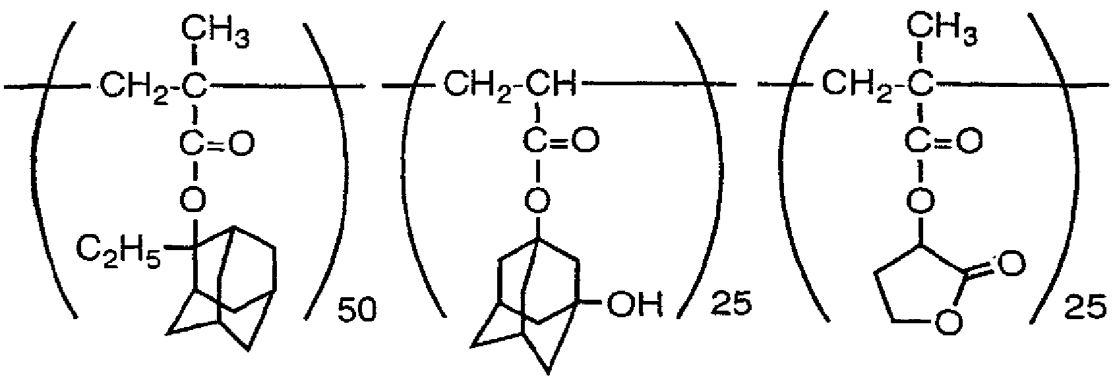
Resin Synthesis Example 8 (synthesis of resin H)

[0043] 2-ethyl-2-adamantyl methacrylate, 3-hydroxy-1-adamantyl methacrylate and methacrylonitrile were charged in a mole ratio of 5 : 2.5 : 2.5 (20.0g : 9.5g : 2.9g), respectively. Then, tetrahydrofuran was added in an amount of 2 weight times based on the total amount of monomers to prepare a solution. As an initiator, azobisisobutyronitrile was added thereto in an amount of 2 mol% based on the total amount of monomers, followed by heating at 65 °C for about 12 hours. Thereafter, the operation of pouring the reaction solution into a large amount of heptane to cause precipitation was repeated 3 times, thus the solution was purified. As a result, a copolymer represented by the following formula, and having a composition mole ratio of each unit of 50 : 25 : 25, and having a weight-average molecular weight of about 14,000 was obtained. The resulting copolymer is referred to as resin H.



Resin Synthesis Example 11 (synthesis of resin J)

[0046] 2-ethyl-2-adamantyl methacrylate, 3-hydroxy-1-adamantyl methacrylate and α-methacryloyloxy-γ-butyrolactone were charged in a mole ratio of 5.0 : 2.5 : 2.5 (20.0g : 8.9 g : 6.8 g), respectively. Then, methyl isobutyl ketone was added in an amount of 2 weight times based on the total amount of monomers to prepare a solution. As an initiator, azobisisobutyronitrile was added thereto in an amount of 2 mol% based on the total amount of monomers, followed by heating at 85 °C for about 5 hours. Thereafter, the operation of pouring the reaction solution into a large amount of heptane to cause precipitation was repeated 3 times, thus the solution was purified. As a result, a copolymer represented by the following formula, and having a composition mole ratio of each unit of 50 : 25 : 25, and having a weight-average molecular weight of about 7,500 was obtained. The resulting copolymer is referred to as resin J.



Examples 1 and 2

[0047] 10 parts of the resin D or E, 0.2 part of p-tolyldiphenylsulfonium trifluoromethanesulfonate ("MDS-205" manufactured by MIDORI Chemical K. K.) as an acid generator, and 0.015 part of 2,6-diisopropylaniline as a quencher were dissolved in 45 parts of 2-heptanone. The resulting solution was filtered through a filter made of a fluorine resin with a pore diameter of 0.2 μm to prepare a resist solution. The resist solution thus prepared was applied onto a silicon wafer (contact angle of water : 50 °) which had been subjected to a treatment with hexamethylsilazane at 23 °C for 20 seconds, and a silicon wafer coated with an organic antireflection film thereon so that the film thickness after drying was 0.5 μm. The organic antireflection film was formed by applying "DUV-42" manufactured by Brewer Co., under the baking conditions of 215 °C for 60 seconds so that the thickness was 570 Å. Prebake after application of the resist solution was conducted under the conditions of 120 °C for 60 seconds on a direct hot plate.

Table 2

Example No.	Resin	Antireflection film	PEB	Effective sensitivity (mj/cm^2)		Resolution (μm)		Adhesion
				Dark	Bright	Dark	Bright	
3	A	DUV-42	120°C	38	36	0.24	0.21	O
4	C	DUV-30-16	120°C	20	20	0.22	0.21	O
5	F	DUV-42	100°C	42	44	0.22	0.18	O
6	G	DUV-42	80°C	24	24	0.20	0.20	O
7	H	DUV-30-16	120°C	32	30	0.21	0.19	O
8	I	DUV-30-16	110°C	22	22	0.22	0.21	O
Comparative 1	X	DUV-42	120°C	26	28	0.24	0.21	x
Comparative 2	X	DUV-30-16	120°C	26	24	0.24	0.21	x

[0051] As shown in Tables 1 and 2, the resists using the resins specified in the present invention do not cause peeling of a pattern at the time of development, and are excellent in adhesion to a substrate. The resolution is also good. Particularly, the resists of Examples 1 and 2, and 4 to 8 using a resin having a unit of 2-ethyl-2-adamantyl methacrylate are improved in resolution as compared with the resists of Comparative Examples. The resist of the present invention is also good in dry etching resistance while not being largely impaired in sensitivity.

Examples 9 to 11 and Comparative Examples 3

[0052] 10 parts of the resin shown in Table 3, 0.2 part of p-tolyldiphenylsulfonium perfluorooctanesulfonate as an acid generator, and 0.0075 part of 2,6-diisopropylaniline as a quencher were dissolved in a mixed solvent of 57 parts of propylene glycol monomethyl ether acetate and 3 parts of γ-butyrolactone. The resulting solution was filtered through a filter made of a fluorine resin with a pore diameter of 0.2 μm to prepare a resist solution. The resist solution thus prepared was applied onto a silicon wafer coated with an organic antireflection film thereon so that the film thickness after drying was 0.39 μm. The organic antireflection film was formed by applying "DUV-30-16" manufactured by Brewer Co., under the baking conditions of 215 °C for 60 seconds so that the thickness was 1600 Å. Prebake after application of the resist solution was conducted for 60 seconds on a direct hot plate at a temperature shown in Table 3.

[0053] The wafer on which a resist film was thus formed was exposed to light through a line-and-space pattern with changing the irradiation amount stepwise by using a ArF excimer stepper ["NSR ArF" manufactured by Nikon Corp., NA = 0.55, σ = 0.6]. After exposure, post exposure bake (PEB) was conducted on a hot plate at the temperatures shown in Table 1 for 60 seconds. Further, paddle development was conducted with a 2.38 % by weight aqueous solution of tetramethylammoniumhydroxide for 60 seconds. The bright field pattern after the development was observed by a scanning electron microscope to determine its effective sensitivity and resolution according to the following methods.

Effective sensitivity: shown by the irradiation amount whereby a 0.18 μm line-and-space pattern is formed at 1 : 1.
Resolution: shown by the minimum dimension of a line-and-space pattern separated at the irradiation amount of the effective sensitivity.

Table 3

Example No.	Resin	Pre-bake Temp.	PEB Temp.	Effective sensitivity (mj/cm^2)	Resolution (μm)
9	J	130°C	115°C	25	0.15
10	C	130°C	130°C	21	0.15
11	A	150°C	140°C	24	0.15

7. A chemical amplifying type positive resist composition which comprises

a resin(2) having a polymerization unit of 2-ethyl-2-adamantyl methacrylate, and a polymerization unit of a monomer selected from 3-hydroxy-1-adamantyl (meth)acrylate, (meth)acrylonitrile, α-methacryloyloxy-γ-butyrolactone and maleic anhydride;
and an acid generator.

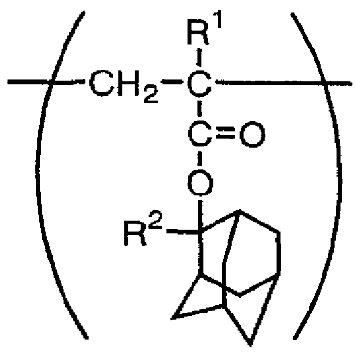
8. A chemical amplifying type positive resist composition according to claim 7 wherein the resin(2) is obtained by copolymerization of monomers which comprise 30 to 80 mol% of 2-ethyl-2-adamantyl (meth)acrylate and 20 to 70 mol% of mixture of 3-hydroxy-1-adamantyl (meth)acrylate, (meth)acrylonitrile, α-methacryloyloxy-γ-butyrolactone and maleic anhydride.

9. A chemical amplifying type positive resist composition according to claim 7 wherein the resin(2) has a polymerization unit of 2-ethyl-2-adamantyl methacrylate and a polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate.

10. A chemical amplifying type positive resist composition according to claim 7 wherein the resin(2) has a polymerization unit of 2-ethyl-2-adamantyl methacrylate, and a polymerization unit of α-methacryloyloxy-γ-butyrolactone.

11. A chemical amplifying type positive resist composition according to claim 10 wherein the resin(2) further has a polymerization unit of 3-hydroxy-1-adamantyl (meth)acrylate.

12. A chemical amplifying type positive resist composition according to claim 7 wherein the resin(2) further has a polymerization unit represented by the following formula (I):



(I)

wherein R^1 represents hydrogen or methyl, and R^2 represents alkyl, provided that the polymerization unit represented by the formula (I) is not the polymerization unit of 2-ethyl-2-adamantyl methacrylate.

13. A chemical amplifying type positive resist composition according to claim 12 wherein the resin(2) is obtained by copolymerization of monomers which comprise 30 to 80 mol% of 2-alkyl-2-adamantyl (meth)acrylate, in which an amount of 2-ethyl-2-adamantyl methacrylate is present and its amount is 20 mol% or more and 20 to 70 mol% of mixture of 3-hydroxy-1-adamantyl (meth)acrylate, (meth)acrylonitrile, α-methacryloyloxy-γ-butyrolactone and maleic anhydride.

14. A chemical amplifying type positive resist composition according to claim 1 and 7 which further comprises an amine as a quencher.

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 982 628 A3

(12) EUROPEAN PATENT APPLICATION

(88) Date of publication A3:
03.05.2000 Bulletin 2000/18

(43) Date of publication A2:
01.03.2000 Bulletin 2000/09

(21) Application number: 99116705.7

(22) Date of filing: 25.08.1999

(51) Int. Cl.[7]: G03F 7/004

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: 26.08.1998 JP 24014398

(71) Applicant:
Sumitomo Chemical Company, Limited
Chuo-ku Osaka 541-8550 (JP)

(72) Inventors:
- Fujishima, Hiroaki
Toyonaka-shi, Osaka (JP)
- Uetani, Yasunori
Toyonaka-shi, Osaka (JP)
- Araki, Kaoru
Kyoto (JP)

(74) Representative:
VOSSIUS & PARTNER
Siebertstrasse 4
81675 München (DE)

(54) A chemical amplifying type positive resist composition

(57) A chemical amplifying type positive resist composition, excellent in adhesion to a substrate and good in resist performances and suitable for exposure using a KrF excimer laser, ArF excimer laser, or the like, which comprises a resin having a polymerization unit of 2-alkyl-2-adamantyl (meth)acrylate and a polymerization unit of a monomer selected from 3-hydroxy-1-adamantyl (meth)acrylate and (meth)acrylonitrile, and an acid generator is provided.

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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